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THE QUANTITATIVE ESTIMATION OF IRON AND COBALT BY THE ROTATING CATHODE.

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THE application of a rotating cathode to the analytical determination of metals by electrolysis was introduced by Gooch in 1903, and the form of apparatus devised, and experiments with the same, are described in several papers from his laboratory.*

Mention is made that a rotating cathode had already been used in the arts for the purpose of securing compact metallic deposits in electroplating.

Soon after the appearance of the first paper the use of a rotating anode for a similar purpose was described by Exner.†

The apparatus used by Gooch and Medway consisted of a small electric motor to the shaft of which, running in a vertical position, was fastened a rubber stopper. An ordinary platinum crucible was slipped on the stopper, electrical contact being made through a strip of platinum connecting with the shaft. A beaker containing the solution to be electrolyzed was raised upon a suitable standard until the crucible was immersed to within eight or nine millimeters of the edge. For anode a piece of platinum foil hung upon the inside of the beaker was used.

The crucible serving as cathode was revolved from 600 to 800 revolutions a minute. The current varied from 0.8 to 4.0 amperes, the corresponding current density per 100 sq. cm. being from 2.7 to 13.3. The time required for a determination was greatly diminished compared with that required in former electrolytic methods in which the ordinary stationary electrodes are used, being, in the case of copper, fifteen minutes on the average, zinc thirty minutes, etc. The results for a large number of experiments shown left nothing to be desired, varying in most cases but one- or two-tenths of a milligram from the correct values.

The metals experimented with, the results of which are shown in the above papers referred to, are copper, silver, nickel, cadmium, tin, zinc, and gold.

* Gooch and Medway, *Am. Jour. Sci.*, XV, 320 (1903). Medway, *ib.*, XVIII, 56, 180 (1904), and *Zen. amorg. Chem.* 35, 414, etc.

† Exner, *Jour. Am. Chem. Soc.*, XXV, 806 (1903).

The experiments reported herewith were carried out with apparatus quite similar to that described above. Copper was first tried, to show whether or not the right conditions had been secured. A solution of copper sulfate several times recrystallized was made, and standardized gravimetrically by a number of closely agreeing determinations made by precipitation with sodium carbonate, igniting and weighing the copper as copper oxid. The results and details are shown in table I. The solution to be electrolyzed was acidified with twelve drops of sulfuric acid 1:4, and occupied a volume of 65 cc.

TABLE I. Copper.

No.	CuSO ₄ taken, cc.	Current, amp.	N. D., 100.	Speed per min.	Time, min.	Cu taken, gm.	Cu found, gm.	Error, gm.
1.....	20 00	1.5	8 9	687	15	0.1110	0.1116	+0.0006
2.....	20.00	1.5	8.9	687	15	0.1110	0.1112	+0.0002
3.....	20 00	1.5	8.9	775	15	0.1110	0.1117	+0.0007
4.....	20 00	1.5	8 9	750	15	0.1110	0.1110	±0.0000
5.....	20.00	1.5	8.9	750	15	0.1110	0.1111	+0.0001
6.....	20.00	1.5	8.9	812	15	0.1110	0.1110	±0.0000
7.....	20 00	1.5	8 9	775	15	0.1110	0.1111	+0.0001
8.....	20 00	1.5	8 9	800	15	0.1110	0.1110	±0.0000
9.....	* 30.00	1.5	8 9	1,000	8	0.0760	0.0760	±0.0000
10.....	25 00	1.5	8.9	875	15	0.0634	0.0634	±0.0000
11.....	25 00	2.2	13.1	800	15	0.0634	0.0635	+0.0001
12.....	25.00	2.7	16.1	850	15	0.0634	0.0634	±0.0000
13.....	25.00	3.0	17.9	825	15	0.0634	0.0635	+0.0001
14.....	25.00	2.8	16.7	850	10	0.0634	0.0634	±0.0000

*A new standard solution made up.

The salt used for the estimating of iron was ferrous ammonium sulfate in ammonium oxalate solution, 1:6, 50 cc. After running for ten minutes the motor was stopped and the sides of the beaker were washed down, as was done with all the experiments with the rotating cathode. It was found that great care was needed in drying the deposit on the crucible by dipping in water, then in alcohol, and holding over a low flame of a Bunsen burner. The deposit was removed by dipping in concentrated hydrochloric acid for a few seconds, and came off in flakes. The completeness of the deposition may be tested at any time toward the end, by removing a drop of the solution, acidifying with hydrochloric acid, and adding potassium sulfocyanide. The results are given in table II.

TABLE II. Iron.

No.	Ferrous am. sulf. taken, gm.	Cur- rent, amp.	N. D., 100.	Speed.	Time, min.	Iron taken, gm.	Iron found, gm.	Error, gm.
1	0.4384	2.0	11.9	800	15	0.0625	0.0631	+0.0006
2	0.4000	2.9	17.3	800	15	0.0561	0.0563	+0.0002
3	0.4000	2.8	16.7	800	18	0.0561	0.0550	-0.0011
4	0.4000	2.6	15.5	800	18	0.0561	0.0561	±0.0000
5	0.4000	2.1	12.5	800	18	0.0561	0.0563	+0.0002
6	0.4000	2.1	12.5	800	20	0.0561	0.0560	-0.0001
7	0.4000	2.1	12.5	800	20	0.0561	0.0561	±0.0000
8	0.4000	2.2	13.1	750	20	0.0561	0.0563	+0.0002
9	0.4000	2.1	12.5	775	20	0.0561	0.0561	±0.0000
10	0.4000	2.9	17.3	800	15	0.0561	0.0563	+0.0002

For experiments in the estimation of cobalt the nitrate was used, the solution being standardized by conversion to the oxid and reducing in a Rose crucible with hydrogen. The results follow:

TABLE III. Cobalt.

No.	Cobalt nitrate sol. taken, cc.	Am- mon- ium oxa- late, gm.	Cur- rent, amp.	N. D., 100.	Speed.	Time, min.	Cobalt taken, gm.	Cobalt found, gm.	Error.
1	25.00	2.5	2.0	11.9	800	32	Lost.
2	25.00	2.0	2.0	11.9	800	35	Lost.
3	25.00	2.0	2.0	11.9	800	32	Lost.
4	25.00	2.0	2.0	11.9	800	35	0.0600	0.0597	-0.0003
5	25.00	2.0	3.0	17.9	800	30	0.0600	0.0598	-0.0002
6	25.00	2.0	2.4	14.3	800	35	0.0600	0.0598	-0.0002
7	25.00	2.0	2.4	14.3	800	33	0.0600	0.0599	-0.0001
8	25.00	2.0	2.5	14.9	800	35	0.0600	0.0605	+0.0005
9	25.00	2.0	2.5	14.9	800	30	0.0600	0.0588	-0.0012
10	25.00	2.0	2.8	16.7	850	25	0.0600	0.0599	-0.0001

These results indicate that, as was to be expected, the application of this method may be extended to include the determination of other metals, and must prove a valuable addition to the processes at the disposal of the analytical chemist on account of its accuracy and rapidity.

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